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(12) **DEMANDE DE BREVET CANADIEN**
CANADIAN PATENT APPLICATION

(13) **A1**

(54) Titre : NOUVEAUX COMPOSES DE PHOSPHONIUM PHOSPHINATE ET ET LEURS PROCEDES DE PREPARATION
(54) Title: NOVEL PHOSPHONIUM PHOSPHINATE COMPOUNDS AND THEIR METHODS OF PREPARATION

$$\left[\begin{array}{cc} R_1 & R_2 \\ & P \\ R_4 & R_3 \end{array} \right]^+ \left[\begin{array}{cc} R_5 & Y_1 \\ & P \\ R_6 & Y_2 \end{array} \right]^- \qquad (I)$$

(57) Abrégé/Abstract:
Novel phosphonium phosphinate compounds and their methods of preparation are disclosed. Phosphonium salts are ionic liquids that are useful as polar solvents. The novel phosphonium phosphinate compounds have the general formula (I): (see formula I) wherein R_1, R_2, R_3, R_4, R_5 and R_6 is independently a hydrogen or hydrocarbyl, unsubstituted or substituted; Y_1 is O or S; and Y_2 is O or S.

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ABSTRACT

Novel phosphonium phosphinate compounds and their
methods of preparation are disclosed. Phosphonium salts are
ionic liquids that are useful as polar solvents. The novel
5 phosphonium phosphinate compounds have the general formula (I):

$$\left[\begin{array}{c} R_1 \diagdown \quad \diagup R_2 \\ P \\ R_4 \diagup \quad \diagdown R_3 \end{array} \right]^{+} \left[\begin{array}{c} R_5 \diagdown \quad Y_1 \\ P \\ R_6 \diagup \quad \diagdown Y_2 \end{array} \right]^{-} \qquad \text{Formula (I)}$$

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wherein R_1, R_2, R_3, R_4, R_5 and R_6 is independently a hydrogen or
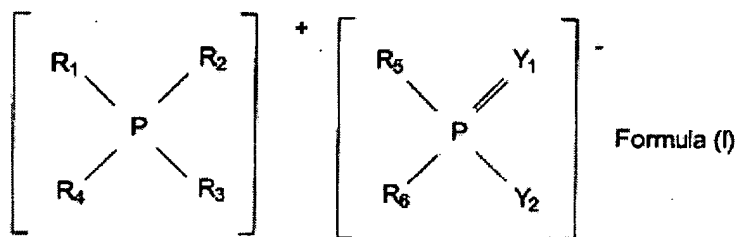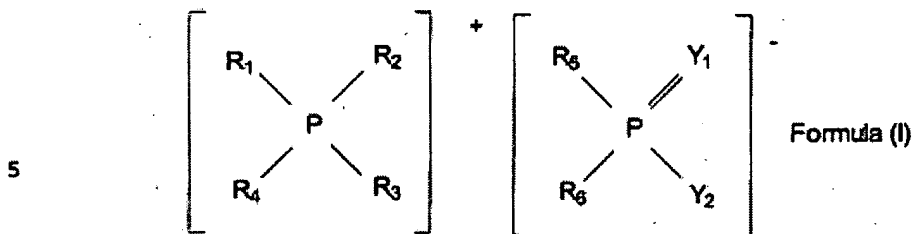hydrocarbyl, unsubstituted or substituted; Y_1 is O or S; and Y_2
is O or S.

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<u>CLAIMS</u>:

1.   A compound of the formula (I):

$$\left[\begin{array}{cc} R_1 & R_2 \\ & P & \\ R_4 & R_3 \end{array}\right]^{+} \left[\begin{array}{cc} R_6 & Y_1 \\ & P & \\ R_6 & Y_2 \end{array}\right]^{-} \quad \text{Formula (I)}$$

wherein:

each of R_1, R_2, R_3, R_4, R_5, and R_6 is independently a hydrogen atom or a hydrocarbyl groups, provided that not more than two of R_1 to R_4 and not more than one of R_5 and R_6 are hydrogen;

Y_1 is O or S; and

Y_2 is O or S.

2.   A compound of claim 1 wherein each of R_1, R_2, R_3, R_4, R_5, and R_6 is independently an alkyl group of 1 to 30 carbon atoms, a cycloalkyl group of 3 to 7 carbons, an alkenyl group of 2 to 30 carbon atoms, an alkynyl group of 2 to 30 carbon atoms, an aryl group of 6 to 18 carbon atoms, or an aralkyl group.

3.   A compound of claim 2 wherein each of R_1, R_2, R_3, and R_4, is independently an alkyl group of 5 to 20 carbon atoms.

4.   A compound of claim 3 wherein each of R_1, R_2, and R_3 is n-hexyl and R_4 is n-tetradecyl.

5.   A compound of any one of claims 1 to 4 wherein each of R_5 and R_6 is

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independently an alkyl group of 1 to 30 carbon atoms, an
alkenyl group of 2 to 30 carbon atoms, an alkynyl group of 2 to
30 carbon atoms, an aryl group of 6 to 18 carbon atoms, or an
aralkyl group.

5   6.   A compound of claim 5 wherein each of R_5 and R_6 is
independently an alkyl group of 5 to 20 carbon atoms.

7.   A compound of claim 1 wherein R_5 and R_6 are 2,4,4'-
trimethylpentyl and Y_1 and Y_2 are O.

8.   A compound of claim 1 wherein R_5 and R_6 are isobutyl
10   and Y_1 and Y_2 are O.

9.   A compound of claim 1 wherein R_5 and R_6 are cyclohexyl
and Y_1 and Y_2 are O.

10.   A compound of claim 1 wherein R_5 and R_6 are isobutyl
and Y_1 and Y_2 are S.

15   11.   A compound of any one of claims 1 to 10 wherein the
total number of carbons in R_1, R_2, R_3, R_4, R_5, and R_6 is 25 or
more.

12.   A compound of any one of claims 1 to 11 wherein the
total number of carbons in R_1, R_2, R_3, R_4, R_5, and R_6 is 40 or
20   more.

13.   A compound of any one of claims 1 to 12 that is
immiscible with water.

14.   A compound of any one of claims 1 to 9 wherein Y_1 and
Y_2 are both O.

25   15.   A compound of any one of claims 1 to 6 and 10,
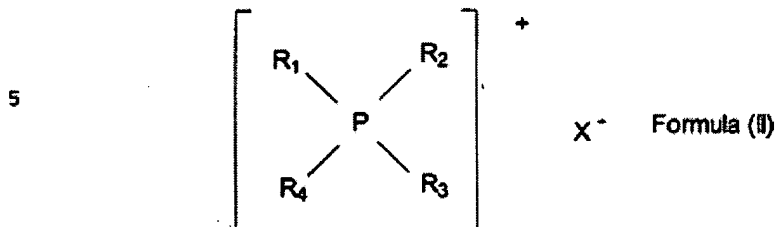wherein Y_1 and Y_2 are both S.

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16.   A method for preparing a phosphonium phosphinate compound of formula (I), as defined in claim 1, wherein:
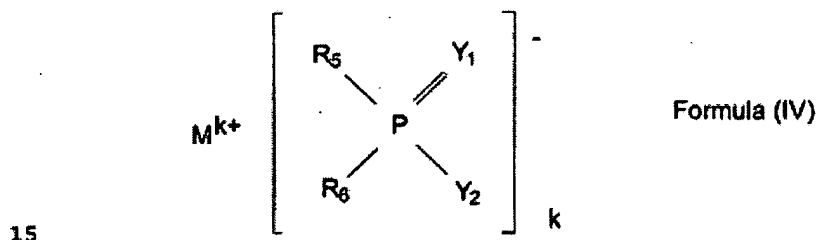
i) a compound of formula (II):

$$\left[\begin{array}{c} R_1 \diagdown \quad \diagup R_2 \\ P \\ R_4 \diagup \quad \diagdown R_3 \end{array} \right]^+ \quad X^- \qquad \text{Formula (II)}$$

wherein R_1 to R_4 are as defined in claim 1, and

X^- is a leaving group,

is reacted with

ii)   a compound of the formula (IV):

$$M^{k+} \left[\begin{array}{c} R_5 \diagdown \quad \diagup\!\!\diagup Y_1 \\ P \\ R_6 \diagup \quad \diagdown Y_2 \end{array} \right]^-_k \qquad \text{Formula (IV)}$$

wherein , R_5, R_6, Y_1 and Y_2 are defined in claim 1, and

M^{k+} is H^+ or a metal cation with valency "k".

17.   The method according to claim 16 wherein M^{k+} is H^+ and X^- is OH^-.

18.   The method according to claim 16 wherein M^{k+} is H^+ and X^- is a leaving group other than OH^- and the reaction is carried out in the presence of a base.

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19.   The method according to claim 16 wherein M^{k+} is a metal cation with valency "k" and X^- is a leaving group other than OH^-.

20.   Use of the compound of any one of claims 1 to 15 as a
5   solvent.

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PATENT AGENTS

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FIELD OF THE INVENTION:

The present invention relates to novel phosphonium phosphinate salts, their methods of preparation and their use, for example, as polar solvents.

5 ## BACKGROUND OF THE INVENTION:

Low melting or liquid phosphonium salts have found utility as polar solvents known as "ionic liquids." Ionic liquids provide an attractive potential alternative to traditional organic solvents for chemical reactions for many

10 reasons. For industrial purposes, the low vapour pressure of ionic liquids is a very important feature. They are essentially non-volatile, a property that eliminates many of the containment problems typically encountered with traditional organic solvents. Since ionic liquids are often composed of

15 poorly coordinating ions, they have the potential to provide a highly polar yet poorly coordinating solvent. Moreover, many of these solvents are immiscible with traditional organic solvents and therefore provide a non-aqueous polar alternative to two-phase systems. Because of their distinctive solvent

20 characteristics, they can be used to bring unusual combinations of reagents into the same phase. A recent review of the properties and uses of ionic liquids is provided in an article entitled "Room-Temperature Ionic Liquids. Solvents for Synthesis and Catalysis," by Thomas Welton (Chem. Rev. 1999,

25 99, 2071-2083).

Ionic liquids provide solvents with a wide liquid range and a high degree of thermal stability. However, there remains a need for increasing the solvent options available to chemists by developing novel ionic liquids with distinctive

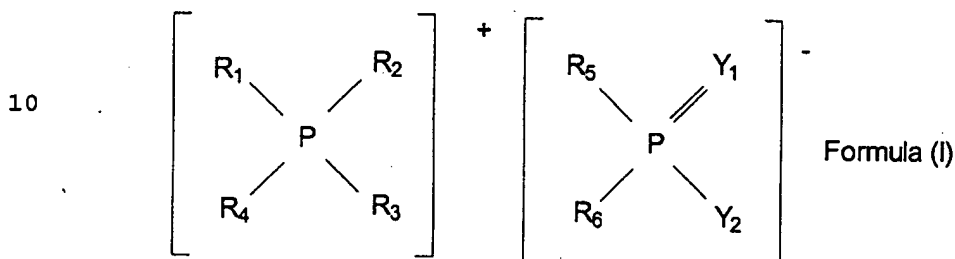30 physical and chemical properties.

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SUMMARY OF THE INVENTION:

The current invention provides novel phosphonium phosphinate compounds and methods of preparing these compounds. The phosphonium phosphinate compounds can have a broad range of
5 phosphonium cations and a broad range of phosphinate and dithiophosphinate anions.

The novel phosphonium phosphinates have the general formula (I):

10

$$
\left[\begin{array}{c} R_1 \\ \diagdown \\ \\ R_4 \end{array} \begin{array}{c} R_2 \\ \diagup \\ P \\ \diagdown \\ R_3 \end{array} \right]^{+} \left[\begin{array}{c} R_5 \\ \diagdown \\ \\ R_6 \end{array} \begin{array}{c} Y_1 \\ \diagup\diagup \\ P \\ \diagdown \\ Y_2 \end{array} \right]^{-} \quad \text{Formula (I)}
$$

wherein:

each of R_1, R_2, R_3, R_4, R_5, and R_6 is independently a
15 hydrogen atom or a hydrocarbyl group, provided that not more than two of R_1 to R_4 and not more than one of R_5 and R_6 are hydrogen;

Y_1 is O or S; and

Y_2 is O or S.

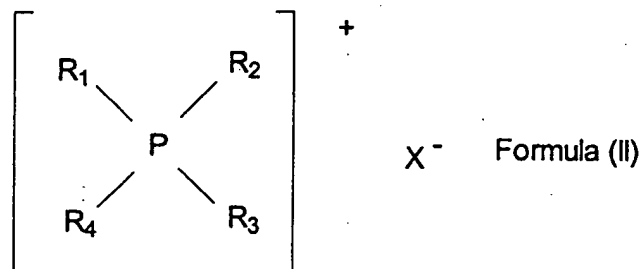20 Preferably, each of R_1 to R_6 is a hydrocarbyl group.

In another aspect, the invention provides a process for preparing a phosphonium phosphinate compound of formula (I), as defined above, wherein:
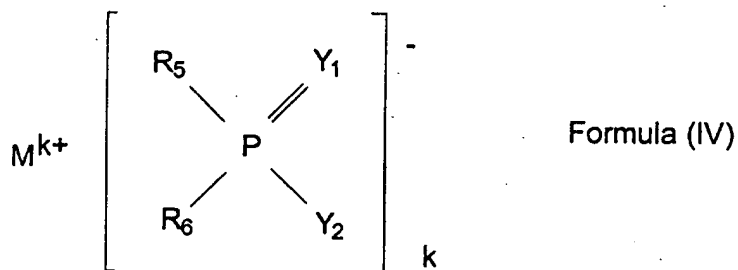
i) a compound of formula (II):

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$$\left[\begin{array}{c} R_1 \\ P \\ R_4 \end{array} \begin{array}{c} R_2 \\ \\ R_3 \end{array} \right]^{+} \quad X^{-} \qquad \text{Formula (II)}$$

5

wherein R_1 to R_4 are as defined above,

and X^- is a leaving group,

is reacted with

ii)   a compound of the formula (IV):

10

$$M^{k+} \left[\begin{array}{c} R_5 \\ P \\ R_6 \end{array} \begin{array}{c} Y_1 \\ \\ Y_2 \end{array} \right]^{-}_{k} \qquad \text{Formula (IV)}$$

wherein , R_5, R_6, Y_1 and Y_2 are as defined in formula
15 (I), and
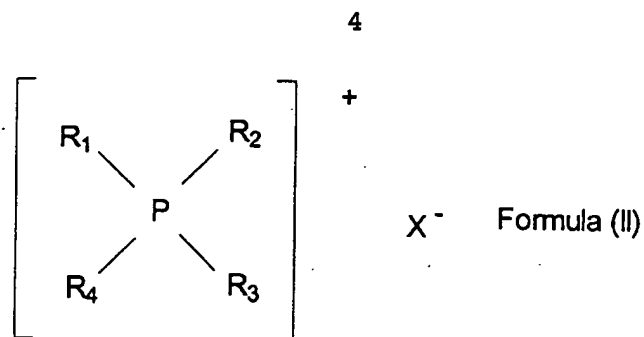
M^{k+} is H^+ or a metal cation with valency "k".
Preferably, if M^{k+} is H^+ and X^- is a leaving group other than
OH^-, then the reaction is carried out in the presence of a base.
A base is not needed if X^- is OH^- and M^{k+} is H^+. If M^{k+} is a
20 metal cation with valency "k", then X^- is a leaving group other
than OH^-.

Thus, in one embodiment, compounds according to
formula (I) can be prepared by reacting:
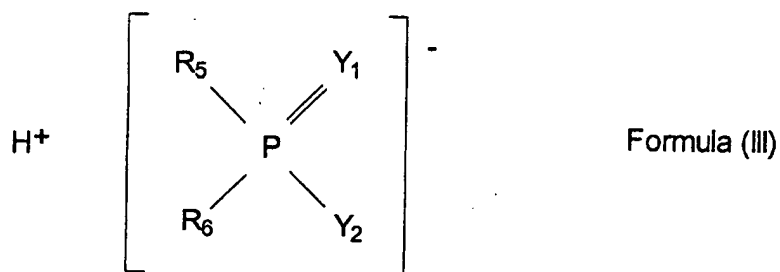
i) a compound of formula (II):

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$$\left[\begin{array}{cc} R_1 & R_2 \\ & P \\ R_4 & R_3 \end{array} \right]^{+} \qquad X^{-} \qquad \text{Formula (II)}$$

wherein R_1 to R_4 are as defined above for formula (I) and

X^- is a leaving group, for example hydroxide (OH^-), acetate, sulfate, or a halide, preferably chloride, bromide or iodide,

with

ii) a compound of formula (III):

$$H^{+} \left[\begin{array}{cc} R_5 & Y_1 \\ & P \\ R_6 & Y_2 \end{array} \right]^{-} \qquad \text{Formula (III)}$$

wherein R_5, R_6, Y_1 and Y_2 are as defined above for formula (I), and

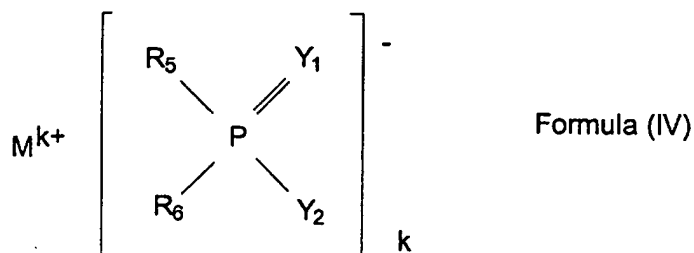when X^- of formula (II) is any leaving group other than OH^-, with

iii) a base, for example a hydroxide or a carbonate of an alkali metal or alkaline earth metal.

In another embodiment, compounds according to formula (I) can also be prepared by reacting a compound of the formula (II), as defined above, with ii) a compound of the formula (IV):

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$$M^{k+} \left[\begin{array}{c} R_5 \diagdown \quad \diagup\!\!\diagup Y_1 \\ P \\ R_6 \diagup \quad \diagdown Y_2 \end{array} \right]_k^{-}$$

Formula (IV)

5   wherein R_5, R_6, Y_1 and Y_2 are as defined above for formula (I), and

M^{k+} is ammonium or a metal and k is the valency of the metal. Appropriate metals are any metals that form water-soluble salts with anions, for example, alkali metals,
10 preferably Na^+ or K^+.

In another aspect of the invention, the compounds according to formula (I) are useful as ionic solvents.

BRIEF DESCRIPTION OF DRAWINGS:

Having generally described the nature of the
15 invention, preferred embodiments will now be described with reference to the accompanying drawings, in which:

Figure 1 shows the results of a TGA assay on trihexyl(tetradecyl)phosphonium bis(2,4,4'-trimethyl-pentyl)phosphinate.

20   Figure 2 shows the results of a TGA assay on trihexyl(tetradecyl)phosphonium diisobutylphosphinate.

Figure 3 is a ^{31}P NMR spectrum of trihexyl(tetradecyl)phosphonium dicyclo-hexylphosphinate.

Figure 4 shows the results of a thermogravimetric
25 analysis (TGA) assay on trihexyl(tetradecyl)phosphonium dicyclohexylphosphinate.

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Figure 5 is a ^{31}P NMR spectrum of trihexyl-
(tetradecyl)phosphonium diisobutyldithiophosphinate.

Figure 6 shows the results of a TGA assay on
trihexyl(tetradecyl)phosphonium diisobutyldithiophosphinate.

5 **DESCRIPTION OF PREFERRED EMBODIMENTS:**

The current invention concerns compounds of the
general formula (I), as defined above, wherein: each of R_1, R_2,
R_3, R_4, R_5, and R_6 is independently a hydrogen or hydrocarbyl
group; Y_1 is O or S; and Y_2 is O or S. It is possible for the
10 groups R_1 to R_6 to bear substituents, or to include heteroatoms,
provided that the substituents or heteroatoms do not interfere
with the preparation of the compounds of the invention, and do
not adversely affect the desired properties of the compound.
Acceptable substituents include alkoxy, alkylthio, acetyl, and
15 hydroxyl groups, and acceptable heteroatoms include oxygen and
sulphur. Substituents are likely to increase the cost of the
compounds of the invention and as the compounds are often used
as solvents, they are used in such volume that cost is a
significant factor. Hence, it is contemplated that, for the
20 most part, substituents will not be present.

Preferably, each of R_1, R_2, R_3, R_4, R_5, and R_6 is
independently an alkyl group of 1 to 30 carbon atoms, a
cycloalkyl group of 3 to 7 carbon atoms, an alkenyl group of 2
to 30 carbon atoms, an alkynyl group of 2 to 30 carbon atoms,
25 an aryl group of 6 to 18 carbon atoms, or an aralkyl group.

Alkyl groups that exceed 18 carbon atoms, especially
those that exceed 20 carbon atoms, are likely to increase
costs. Since cost is a significant factor in producing a
solvent, it is contemplated that, for practical purposes, the
30 alkyl groups will typically not exceed 20 carbon atoms.
Therefore, more preferred are compounds according to formula

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(I) wherein each of R_1, R_2, R_3, R_4, R_5, and R_6, is independently an alkyl group of 5 to 20 carbon atoms. For example, R_1, R_2, R_3, R_4, R_5, and R_6 may be n-butyl, isobutyl, n-pentyl, cyclopentyl, isopentyl, n-hexyl, cyclohexyl, (2,4,4'-

5   trimethyl)pentyl, cyclooctyl, tetradecyl, etc., although it is preferred that at least one of R_1 to R_4 contains a higher number of carbon atoms, for example 14 or more. In many cases, it is desired that R_1 to R_4 shall not be identical. For many purposes, it is desired that at least one of R_1 to R_4 shall

10   contain a significantly higher number of carbon atoms than the others of R_1 to R_4. Compounds in which R_1 to R_4 are not identical are referred to as asymmetric.

For many applications, it will be preferred that Y_1 and Y_2 are both O. For example, in chemical reactions that

15   utilize certain metal catalysts such as Pd(OAc)$_2$, it will be preferred that Y_1 and Y_2 are both O because the presence of thio groups in the phosphinate anion may interfere with the action of the catalyst. Phosphonium thiophosphinate compounds find utility as solvents for chemical reactions that do not involve

20   metal catalysts.

Preferred compounds include compounds according to formula (I) wherein each of R_1, R_2, R_3, R_4, R_5, and R_6, is independently an aryl group or substituted aryl group. For example, one or more of R_1, R_2, R_3, R_4, R_5, and R_6 may be phenyl,

25   phenethyl, xylyl, or naphthyl.

For some purposes, compounds according to formula (I) that are hydrophobic or "water immiscible" are preferred. The term "water immiscible" is intended to describe compounds that form a two phase system when mixed with water but does not

30   exclude ionic liquids that dissolve in water nor ionic liquids that will dissolve water, provided that the two phase system forms. Therefore, compounds that have a large total number of

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carbons, equal to or greater than 20 and in particular greater than 25 or 26, or have at least one aryl group are preferred because they are more hydrophobic. Water immiscibility is a desirable feature of phosphonium phosphinates not only because

5   it renders the compounds useful for biphasic reactions with an aqueous phase, but also because it facilitates purification and isolation of the phosphonium phosphinate when prepared according to certain methods. There is no critical upper limit on the total number of carbon atoms that may be present in R_1 to

10   R_6. However, it is unlikely that the total will exceed 50.

For many purposes, a material that is a liquid at room temperature is very valuable. Preferred compounds, therefore, are those in which the particular groups R_1 to R_6 are selected to yield compounds that are liquid at room

15   temperature. Selection of particular values for R_1 to R_6 to achieve particular melting points and degrees of water immiscibility is within the competence of a person skilled in the art, although it may require some routine experimentation. For example, the degree of asymmetry and branching of the

20   hydrocarbyl groups R_1 to R_6 of the phosphonium cation or phosphinate anion are important determinants of the melting point: the melting point tends to decrease as the degree of asymmetry and branching is increased. Branching can occur at the alpha or omega carbon or at any intermediate point.

25   Increasing the total number of carbon atoms present in the hydrocarbyl groups R_1 to R_6 will tend to increase the melting point, although this effect will be counteracted somewhat by assymetry and branching. For instance, the properties of compounds whose phosphonium cation contains four decyl groups

30   as R_1 to R_4 will differ from those of a compound having three undecyl groups and one heptyl, despite the fact that both cations have a total of 40 carbon atoms.

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For some purposes, compounds according to formula (I)
that have chirality may be especially preferred, as they
further provide a chiral environment for chemical reactions.
Examples include compounds in which one of R_1 to R_6 is an

5   enantiomer of 2,4,4'-trimethylpentyl, which has one chiral atom.

Examples of preferred compound according to formula
(I) include those in which:

each of R_1, R_2, and R_3 is n-hexyl and R_4 is n-
tetradecyl and

10   R_5 and R_6 are 2,4,4'-trimethylpentyl and Y_1 and Y_2 are
O; or

R_5 and R_6 are isobutyl and Y_1 and Y_2 are O; or

R_5 and R_6 are cyclohexyl and Y_1 and Y_2 are O; or

R_5 and R_6 are isobutyl and Y_1 and Y_2 are S.

15   The current invention also provides methods for
preparing the phosphonium phosphinate compounds according to
formula (I). In general, phosphonium phosphinates can be
prepared by reacting a phosphonium salt of formula (II) with
either: 1) a phosphinic acid of formula (III) and a base, or 2)
20   a phosphinate salt of formula (IV). Alternatively, phosphonium
phosphinates can be prepared by reacting a phosphonium
hydroxide of formula (II) with a phosphinic acid. The
temperature of the reaction is not critical, but the reaction
is conveniently done at elevated temperature, up to about
25   100° C, preferably in the range of 45-70° C. Use of a higher
temperature facilitates phase separation.

10

If a phosphonium phosphinate is immiscible with water, it can be prepared by first mixing a phosphonium salt of formula (II) with a phosphinic acid compound of formula (III) and water, with stirring or other means of mixing, then adding

5   a base. The mixture is stirred for an additional period. When mixing is stopped, the reaction mixture will separate into an organic phase that contains the phosphonium phosphinate product and an aqueous phase. The aqueous phase can be decanted, and the organic phase can then be washed with water to remove the

10   salt byproducts formed by the reaction (for example, sodium chloride). If desired, residual water can be removed from the organic layer by, for example, vacuum-stripping.

In a variation of the method described above, the phosphonium salt and water are mixed together first, then

15   sodium hydroxide is added, and the phosphinic acid is added last. When mixing is stopped, the reaction mixture will separate into an aqueous phase and organic phase that can be processed further as described in the method above.

Phosphonium phosphinates according to formula (I)

20   that are immiscible with water can also be prepared by mixing a phosphonium salt of formula (II) with a phosphinate salt of formula (IV) and water, with stirring. The mixture is stirred for an additional period, say one hour. When mixing is stopped, the reaction mixture will separate into aqueous and

25   organic layers. The aqueous layer can be decanted, and the organic layer can be washed several times with water, to remove any remaining $[M^+] [X^-]_k$. If desired, dissolved water can be removed from the organic layer by, for example, vacuum-stripping.

30   Another preferred method can be used to prepare phosphonium phosphinates that are either miscible or immiscible with water. Phosphonium phosphinates according to formula (I)

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can be prepared by reacting a phosphonium hydroxide of formula (II), i.e. a compound of formula (II) in which X^- is OH^-, with a phosphinic acid of formula (III) to produce a phosphonium phosphinate and water. The water produced by this acid-base

5   reaction can be removed by, for example, vacuum-stripping. Since phosphonium phosphinates produced by this method do not have to be washed with water to remove salt, this method can be used to prepare phosphonium phosphinates that are either miscible or immiscible with water. This method is preferred

10   for preparing phosphonium phosphinates that have a small total number of carbons, of the order of 7 to 10 carbons.

It should be noted that the compounds of formula (I) contain up to six hydrocarbyl groups R_1 to R_6. The particular properties of a compound of formula (I) depend upon the values

15   taken by these six groups. Selection of different values for these groups therefore permits fine tailoring of the properties of the compound of the invention. Hence compounds can be designed to be liquid at a particular temperature and to be water-immiscible. Change in the value of one or more of the

20   groups R_1 to R_6 can effect change in these properties. The presence of six groups for this purpose is advantageous when compared with known ionic liquids based on dialkylimidazolium cations, which have only two groups that can be varied.

For the most part compounds of the invention have a

25   density less than 1. Consequently, they form the upper phase of two phase systems with water. In this respect, they differ from known ionic liquids based on dialkyl imidazolium cations, which tend to have a density greater than 1 and therefore form the lower phase of two phase systems with water.

30   The phosphonium phosphinate salts of the current invention may be used as polar solvents. In a preferred embodiment, phosphonium phosphinates of the current invention

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can be used as polar solvents for chemical reactions such as
Michael additions, aryl coupling, Diels-Alder, alkylation,
biphasic catalysis, Heck reactions, hydrogenation, or for
enzymatic reactions, for example lipase reactions.

5   In the following examples (see Examples 5 and 6), it
will be shown that the phosphonium phosphinates of the current
invention are suitable solvents for the synthesis of biphenyl
via homo-coupling of bromobenzene or iodobenzene. Biaryls are
of great importance in synthetic organic chemistry, as they
10   have found many industrial and pharmacological applications.
Elaboration of liquid crystals, for example, often relies on
the synthesis of a biaryl framework. Among the natural
products biophenomycin and steganacin posses this biaryl
molecular substructure. Hence, their production in a cost-
15   effective fashion is especially important. The Ullman
Synthesis of biaryls typically demands high temperature
conditions (200° C) and requires equimolar amounts of copper.
The use of palladium catalysts and an appropriate ionic
solvent, such as the phosphonium phosphinates of the current
20   invention, may avoid the need for stoichiometric amounts of
metal and high temperature.

        In Example 8, the suitability of the phosphonium
phosphinates as solvents for palladium catalyzed carbonylation
is demonstrated. The palladium catalyzed carbonylation
25   reactions of aryl-X derivatives constitute a powerful method of
C-C coupling reaction for the synthesis of various aromatic
carboxylate acid derivatives such as amides and esters. The
aryl palladium species formed as the intermediate undergoes the
facile CO insertion, followed by the nucleophilic attack of
30   alcohol, water, and amines to give the acid, esters and amides
respectively. This reaction can be carried out using aryl
halide with carbon monoxide and a nucleophile in the presence
of catalytic amount of a palladium compound. Other metal

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catalysts derived from Co and Ni have also been used as catalysts.

As can be seen from the following examples, some of which are in accordance with the invention and some of which
5 are comparative, yields vary with choice of solvent in the various reactions. In many of the examples, the phosphonium phosphinates of the present invention give good, or best, results. Hence they provide a valuable enhancement in methods of synthesis.

10 **EXAMPLES:**

Example 1:

trihexyl(tetradecyl)phosphonium bis(2,4,4'-trimethylpentyl)-phosphinate

Trihexyl(tetradecyl)phosphonium bis(2,4,4'-trimethyl-
15 pentyl)phosphinate was prepared according to the following method. A 5 liter stirred jacketed reactor was charged with:

1.880 moles of trihexyl(tetradecyl)phosphonium chloride

(1003 g of CYPHOS 3653 containing

20   97.2 % trihexyl(tetradecyl)phosphonium chloride)

1.875 moles of bis(2,4,4'-trimethylpentyl)phosphinic acid

(625 g of CYANEX 272 containing

25   87 % bis(2,4,4'-trimethylpentyl)phosphinic acid)

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and 1369 g of water.

After heating the mixture to 54° C, a 25 % aqueous
sodium hydroxide solution (82.3 g of 97 % sodium hydroxide (two
moles), 236.8 g water) was added over about 32 minutes. The
5   two phases were stirred for an additional ½ hr at 55° C.

The agitation was then turned off and the reaction
mixture was allowed to separate into a two-phase system
consisting of an upper organic phase and a lower aqueous phase
(phase separation took about 2 minutes). The lower aqueous
10   phase was decanted and the upper organic layer was washed three
times with about 1300 g of distilled water per wash, by
stirring for one hour at 55° C. The amount of time required for
bulk phase separation increased with each successive wash:
2 minutes, 30 minutes, then for the final wash, 8 minutes was
15   required to achieve bulk separation but 22 hours was required
for the organic layer to clear.

After washing and allowing the phases to separate,
the organic layer was then vacuum stripped to remove dissolved
water. Approximately 230 g of water (13.2 % by weight) was
20   removed after vacuum stripping to 125° C under 4 mmHG pressure.
The final organic layer was perfectly clear.

Results

The aqueous phase was analyzed for chloride ion (see
Table 1). The first decant removed 82.9 % of the chloride ion.
25   The first, second, and third washes additionally removed
12.0 %, 1.7 %, and 0.2 % of the chloride ion, respectively, for
a total of 96.7 %.

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Samples of trihexyl(tetradecyl)phosphonium
bis(2,4,4'-trimethylpentyl) phosphinate were analyzed by TGA
and found to be thermally stable up to approximately 300° C (see
Figure 1). This analysis also indicated that the product may
5 contain as much as 4-6 % moisture.

Table 1

Preparation of trihexyl(tetradecyl)phosphonium bis (2,4,4'-trimethylpentyl)phosphinate

bis(2,4,4'-trimethylpentyl) phosphinic acid (CYANEX 272)	87.0% $R''_2P(O)OH$	R''=bis 2,4,4'-trimethylpentyl
CYPHOS 3653	97.2% $R_3R'PCl$	R=n-butyl
	2.1% R_3PCl	R'=n-tetradecyl
	0.2% HCl	

	Weight (g)	Moles	% Cl	Chloride Balance % Cl Moles Cl	Removed
CYANEX 272	625	1.875		0.000	
CYPHOS 3653	1003	1.880		1.946	
97% NaOH	82.3	1.996		0.000	
Water	1605				

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1st decant	1404	4.0720	1.613	82.9
2nd decant	1283	0.6450	0.233	12.0
3rd decant	1305	0.0873	0.032	1.7
4th decant	1303	0.0096	0.004	0.2
Total			1.882	96.7

Example 2: trihexyl(tetradecyl)phosphonium diisobutylphosphinate

Trihexyl(tetradecyl)phosphonium diisobutylphosphinate
5 was prepared according to the following method. A 5 liter
stirred jacketed reactor was charged with:

2.004 moles of trihexyl(tetradecyl)phosphonium
chloride

(1069 g of CYPHOS 3653)

10   2.004 moles of diisobutylphosphinic acid

(390.8 g of 91.3 % diisobutylphosphinic acid) and

1114 g of water

After heating the mixture to 55° C, a 25 % aqueous
solution of sodium hydroxide (87.7 g of 97 % sodium hydroxide
15 (two moles), 240 g water) was added over 30 minutes. The two
phases were stirred for an additional 1 hr at 55° C.

17

The agitation was then turned off and the reaction mixture was allowed to separate into a two-phase system consisting of an upper organic phase and a lower aqueous phase (phase separation took about 2 minutes). The lower aqueous
5   phase was decanted and the upper organic layer was washed three times each with about 1300 g of distilled water by stirring for one hour at 55° C. The amount of time required for bulk phase separation increased with each successive wash. After washing, the organic and liquid phases were cloudy initially but cleared
10 upon standing overnight.

After washing and allowing the phases to completely separate, the organic layer was then vacuum stripped to remove dissolved water. Approximately 13.4 % water by weight was removed after vacuum stripping to 135° C under 4 mmHG pressure.
15 The final organic layer was perfectly clear.

Results

The decanted aqueous phases were analyzed for chloride ion (see Table 2). The first decant removed 63.7 % of the chloride ion. The first, second, and third washes
20 additionally removed 5.3 %, 0.3 %, and 0.2 % of the chloride ion, respectively, for a total of 69.5 %.

Samples of trihexyl(tetradecyl)phosphonium diisobutylphosphinate were analyzed by TGA and found to be thermally stable up to approximately 300° C (see Figure 2).
25 This analysis also indicated that the product may contain as much as 4-6 % moisture.

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Table 2

Preparation of trihexyl(tetradecyl)phosphonium diisobutylphosphinate

Diisobutylphosphinic acid	91.3% $R''_2P(O)OH$	R''=isobutyl
CYPHOS 3653	97.2% $R_3R'PCl$	R=n-butyl
	2.1% R_3PCl	R'=n-tetradecyl
	0.2% HCl	

	Weight (g)	Moles	% Cl	Chloride Balance Moles Cl	% Cl Removed
$R''_2P(O)OH$	390.8	2.004		0.000	
CYPHOS 3653	1069	2.004		2.074	
97% NaOH	87.7	2.127		0.000	
Water	1354				
1st decant	1245		3.7610	1.321	63.7

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2nd decant	1278	0.3051	0.110	5.3
3rd decant	1270	0.0174	0.006	0.3
4th decant	1300	0.0090	0.003	0.2
Total			1.440	69.5

Example 3: trihexyl(tetradecyl)phosphonium dicyclohexylphosphinate

Trihexyl(tetradecyl)phosphonium
5 dicyclohexylphosphinate was prepared according to the following
method. A stirred jacketed reactor was charged with:

1.907 moles of trihexyl(tetradecyl)phosphonium
chloride

(1017 g of CYPHOS 3653)

10 1.909 moles of di cyclohexylphosphinate

(349 g of di cyclohexylphosphinate) 2.417 moles) and

1200 g of water

After heating the mixture to 55° C, a 25 % aqueous
solution of sodium hydroxide (83.4 g sodium hydroxide
15 (2.022 moles), 210 g water) was added over 30 minutes. The two
phases were stirred for an additional 1 hr at 55° C.

The agitation was then turned off and the reaction
mixture was allowed to separate into a two-phase system
consisting of an upper organic phase and a lower aqueous phase

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(phase separation took about 4 minutes). The lower aqueous phase was decanted and the upper organic layer was washed three times by stirring with 1300 g of distilled water.

After washing and allowing the phases to separate,
5 the organic layer was then vacuum stripped to remove dissolved water. Approximately 210 g of water (14 % water by weight) was removed after vacuum stripping to 138° C under 4 mmHG pressure. The final organic layer was perfectly clear.

Results

10   The decanted aqueous phases were analyzed for chloride ion. The first decant removed 82.2 % of the chloride ion, and the first, second, and third washes additionally removed 14.7 %, 1.7 %, and 0.2 % of the chloride ion, respectively, for a total of 98.8 % (see Table 3).

15   A sample of the organic layer was analyzed by ^{31}P NMR (Figure 3). The NMR spectrum indicates two distinct signals: +33.46 ppm for the phosphonium cation and +31.2741 ppm for the phosphinate anion, consistent with the reaction product trihexyl(tetradecyl)phosphonium di cyclohexylphosphinate.

20   Samples of trihexyl(tetradecyl)phosphonium di cyclohexylphosphinate were analyzed by TGA and found to be thermally stable up to approximately 300° C (see Figure 4). This analysis also indicated that the product may contain as much as 7-8 % moisture.

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Table 3

Preparation of trihexyl(tetradecyl)phosphonium dicyclohexylphosphinate

Dicyclohexylphosphinic acid	98.0% $R''_2P(O)OH$	R''=cyclohexyl
CYPHOS 3653	97.2% $R_3R'PCl$	R=n-butyl
	2.1% R_3PCl	R'=n-tetradecyl
	0.2% HCl	

	Weight (g)	Moles	% Cl	Chloride Balance Moles Cl	% Cl Removed
$R''_2P(O)OH$	439	1.909		0.000	
CYPHOS 3653	1017	1.907		1.973	
97% NaOH	83.4	2.022		0.000	
Water	1200				
1st decant	1249		4.6040	1.622	82.2
2nd decant	1344		0.7650	0.290	14.7

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3rd decant	1300		0.0898	0.033	1.7
4th decant	1310		0.0090	0.003	0.2
Total				1.948	98.8

Table 4

Water Stripping from Phosphonium Phosphinates $R_3R'P \ R''P(O)O$

R''	% Water Removed	Temperature (°C)	Pressure (mmHg)
2,4,4'-trimethylpentyl	13.2	125	4
isobutyl	13.4	135	4
cyclohexyl	14.0	138	4

Example 4: trihexyl(tetradecyl)phosphonium diisobutyl dithiophosphinate

Trihexyl(tetradecyl)phosphonium diisobutyl dithiophosphinate was prepared according to the following method. A stirred jacketed reactor was charged with:

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1.91 moles of trihexyl(tetradecyl)phosphonium chloride

(1019 g of CYPHOS 3653)

1.99 moles of sodium diisobutyldithiophosphinate
5   (925 g of AEROPHINE 3418A, a 50% aqueous solution of sodium diisobutyldithiophosphinate) and

1500 g of water

This mixture was heated to 50° C and stirred for 30 minutes. The agitation was then turned off and the reaction
10   mixture was allowed to separate into a two-phase system consisting of an upper organic phase and a lower aqueous phase (phase separation took about 4 minutes). The lower aqueous phase was decanted and the upper organic layer was washed three times by stirring with 1400 g of distilled water at 50° C.

15   The organic layer was then vacuum stripped to 125° C at 1.2 mmHg pressure. Only 25 g of water was removed. The final organic layer was perfectly clear.

Results

The final product was a liquid at room temperature.
20   The chloride content was 0.0099 %.

The ^{31}P NMR spectrum indicated two distinct signals: +33.37 ppm, phosphonium cation; and +65.81 ppm, dithiophosphinate anion (Figure 5).

Samples of trihexyl(tetradecyl)phosphonium
25   diisobutyldithiophosphinate were analyzed by TGA and found to be thermally stable up to approximately 270° C (see Figure 6).
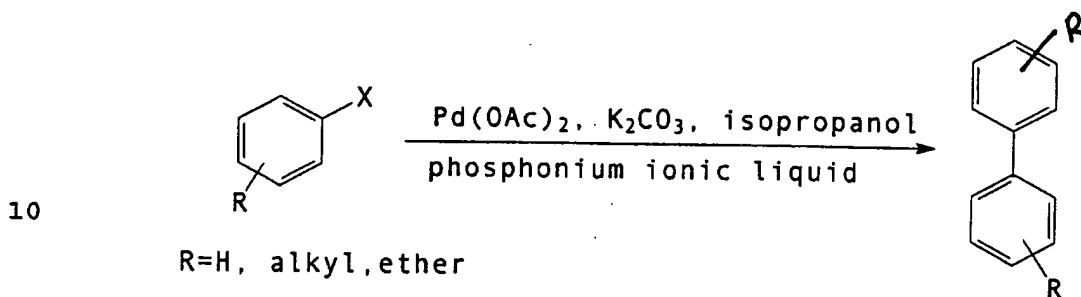
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This analysis also indicated that the product contains approximately 0.5 % water.

Example 5: Biphenyl synthesis via homo-coupling of bromobenzene using Pd(OAc)₂ in various phosphonium ionic liquids

5   In this series of experiments, the homo-coupling of bromobenzene was carried out in various phosphonium ionic liquids.   The reaction proceeds according to:

10   R=H, alkyl,ether

       In the following experiments, a stirred jacketed reactor was charged with:

       1.0 g bromobenzene

15   2.0 g isopropyl alcohol

       1.5 g K₂CO₃

       0.03 g of Pd(OAc)₂

       The reagents were heated at 120° C for 16 hours in a phosphonium ionic liquid solvent selected from the group
20 consisting of:

       trihexyl(tetradecyl)phosphonium dicyclohexylphosphinate;

       trihexyl(tetradecyl)phosphonium decanoate;

       trihexyl(tetradecyl)phosphonium bis-(2,4,4'-trimethylpentyl)phosphinate;

25

trihexyl(tetradecyl)phosphonium triflamide; and

trihexyl(tetradecyl)phosphonium triflate.

The reaction mixture was allowed to cool, poured into
50 ml water, and the total reaction mixture was extracted with
5 petroleum ether (at 45-60° C). The ionic liquid formed a middle
layer that could be recovered. The petroleum ether layer was
washed with water, then with brine, and then concentrated. The
residue was distilled to obtain the required biaryl compound.

Overall yield of biphenyl using the different
10 solvents was evaluated and is tabulated in Table 5. The yield
of biphenyl varied considerably with choice of solvent.
However, the two experiments in which phosphonium phosphinate
compounds were used as solvents provided substantially higher
yields than comparative experiments in which phosphonium
15 triflamide or triflate were used as solvents, thus
demonstrating the suitability of the compounds of the current
invention for use in biphenyl synthesis via homo-coupling of
bromobenzene using Pd(OAc)$_2$. Reaction conditions were not
optimized for any particular solvent, and it is reasonable to
20 expect that overall yields could be improved.

25

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Table 5

Biphenyl synthesis via homo-coupling of bromobenzene using Pd(OAc)2 in various phosphonium ionic liquids

Exp.No.	Ionic Liquid	% Yield
1	trihexyl(tetradecyl)phosphonium dicyclohexylphosphinate	61
2	trihexyl(tetradecyl)phosphonium decanoate	100
3	trihexyl(tetradecyl)phosphonium bis-(2,4,4-trimethylpentyl)phosphinate	52
4	trihexyl(tetradecyl)phosphonium triflamide	26
5	trihexyl(tetradecyl)phosphonium triflate	9

Example 6: Biphenyl synthesis via homo-coupling of iodobenzene in various ionic liquids using Pd(OAc)$_2$ as catalyst

The following reaction is analogous to the reaction in Example 5, except that it is performed using iodobenzene as a starting material.

A stirred jacketed reactor was charged with:

1.0 g iodobenzene

1.5 g isopropyl alcohol

1.5 g K$_2$CO$_3$

0.03 g of Pd(OAc)$_2$

The reagents were heated at 120° C for 18 hours in a phosphonium ionic liquid solvent selected from the group consisting of:

trihexyl(tetradecyl)phosphonium chloride;

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trihexyl(tetradecyl)phosphonium triflate;

trihexyl(tetradecyl)phosphonium triflamide;

trihexyl(tetradecyl)phosphonium bis-(2,4,4'-trimethylpentyl)phosphinate;

5   trihexyl(tetradecyl)phosphonium dicyclohexylphosphinate;

trihexyl(tetradecyl)phosphonium diisobutylphosphinate;

trihexyl(tetradecyl)phosphonium decanoate;

trihexyl(tetradecyl)phosphonium tetrafluoroborate; and

trihexyl(tetradecyl)phosphonium hexaflurophosphate.

10   Overall yield of biphenyl using the different
solvents was evaluated and is tabulated in Table 6. The yield
of biphenyl varied considerably with choice of solvent. Use of
trihexyl(tetradecyl)phosphonium chloride as solvent results in
a particularly low yield of biphenyl because chloride ions
15   interfere with the $Pd(OAc)_2$ catalyst. When phosphonium
phosphinate compounds according to the current invention are
used as solvents, the reaction proceeds with high yields, on
the order of about 100 %. These results demonstrate that
phosphonium phosphinate compounds do not interfere with the
20   $Pd(OAc)_2$ catalyst and therefore are suitable solvents for
biphenyl synthesis via homo-coupling of iodobenzene using
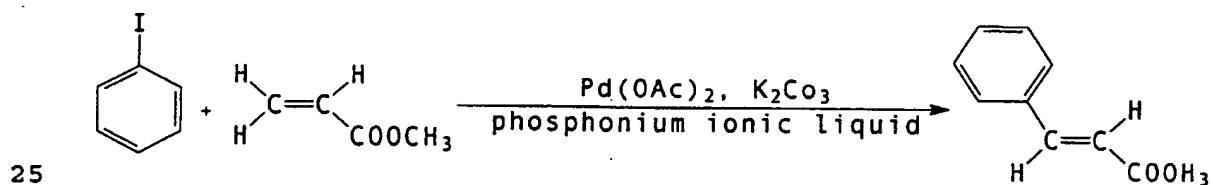$Pd(OAc)_2$ as a catalyst.

25

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Table 6

Homo-coupling of iodobenzene in various ionic liquids using Pd(OAc)2 catalyst

Exp.No.	Ionic Liquid	% Conversion
1	trihexyl(tetradecyl)phosphonium chloride	13
2	trihexyl(tetradecyl)phosphonium triflate	100
3	trihexyl(tetradecyl)phosphonium triflamide	72
4	trihexyl(tetradecyl)phosphonium bis-(2,4,4-trimethylpentyl)phosphinate	78
5	trihexyl(tetradecyl)phosphonium dicyclohexylphosphinate	100
6	trihexyl(tetradecyl)phosphonium diisobutylphosphinate	100
7	trihexyl(tetradecyl)phosphonium decanoate	100
8	trihexyl(tetradecyl)phosphonium tetrafluoroborate	56
9	trihexyl(tetradecyl)phosphonium hexafluorophosphate	50

Example 7: Heck coupling of iodobenzene and methylacrylate in various ionic liquids using Pd(OAc)₂ catalyst

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A stirred jacketed reactor was charged with:

1.0 g iodobenzene

0.86 g methylacrylate

2.0 g K₂CO₃

5   0.05 g of Pd(OAc)₂

The reaction mixture was heated at 80° C for 14 hours in 2.0 g of a phosphonium ionic liquid solvent selected from the group consisting of:

trihexyl(tetradecyl)phosphonium chloride;

10   trihexyl(tetradecyl)phosphonium triflate;

trihexyl(tetradecyl)phosphonium triflamide;

trihexyl(tetradecyl)phosphonium bis-(2,4,4'-trimethylpentyl)phosphinate;

trihexyl(tetradecyl)phosphonium dicyclohexylphosphinate;

15   trihexyl(tetradecyl)phosphonium diisobutylphosphinate;

trihexyl(tetradecyl)phosphonium decanoate;

trihexyl(tetradecyl)phosphonium tetrafluoroborate; and

trihexyl(tetradecyl)phosphonium hexaflurophosphate.

Overall yield is tabulated in Table 7. The yield of
20 the reaction varied considerably with choice of solvent.
Yields on the order of about 100 % were obtained using three of
the phosphonium phosphinate solvents of the current invention,
thus demonstrating that phosphonium phosphinate compounds
provide suitable solvents for Heck coupling reactions.

30

An analogous series of experiments was performed
using ethylacrylate instead of methylacrylate. The results of
these experiments are tabulated in Table 8. In general, the
Heck coupling of iodobenzene and ethylacrylate gave lower
5 yields than the coupling of iodobenzene with methylacrylate.
However, it is clear from these experiments that the
phosphonium phosphinate compounds of the current invention
provide suitable solvents for this Heck coupling reaction.

Table 7

10   **Heck coupling of iodobenzene and methylacrylate in various
ionic liquids using Pd(OAc)2 catalyst**

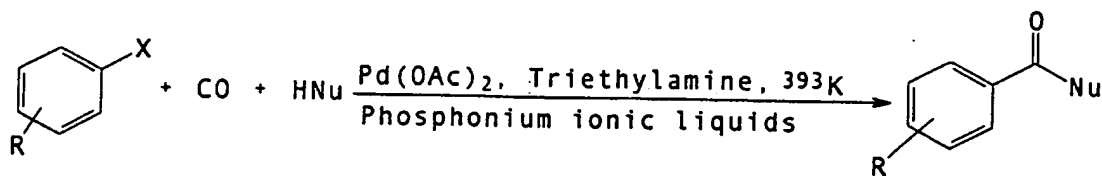| Exp.No.	Ionic Liquid	% Conversion
1	trihexyl(tetradecyl)phosphonium chloride	82
2	trihexyl(tetradecyl)phosphonium triflate	92
3	trihexyl(tetradecyl)phosphonium triflamide	62
4	trihexyl(tetradecyl)phosphonium bis-(2,4,4-trimethylpentyl)phosphinate	100
5	trihexyl(tetradecyl)phosphonium dicyclohexylphosphinate	100
6	trihexyl(tetradecyl)phosphonium diisobutylphosphinate	100
7	trihexyl(tetradecyl)phosphonium decanoate	100
8	trihexyl(tetradecyl)phosphonium tetrafluoroborate	60
9	trihexyl(tetradecyl)phosphonium hexafluorophosphate	82

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Table 8

Heck coupling of iodobenzene and ethylacrylate in various ionic liquids using Pd(OAc)2 catalyst

Exp.No.	Ionic Liquid	% Conversion
1	trihexyl(tetradecyl)phosphonium chloride	42
2	trihexyl(tetradecyl)phosphonium triflate	78
3	trihexyl(tetradecyl)phosphonium triflamide	52
4	trihexyl(tetradecyl)phosphonium bis-(2,4,4-trimethylpentyl)phosphinate	84
5	trihexyl(tetradecyl)phosphonium dicyclohexylphosphinate	10
6	trihexyl(tetradecyl)phosphonium diisobutylphosphinate	86
7	trihexyl(tetradecyl)phosphonium decanoate	87
8	trihexyl(tetradecyl)phosphonium tetrafluoroborate	35
9	trihexyl(tetradecyl)phosphonium hexafluorophosphate	76

Example 8: Carbonylation of iodobenzene in various ionic liquids using Pd(OAc)$_2$ catalyst

The general reaction proceeds as follows:

R=H, alkyl   Nu=nucleophile, e.g. OH, OR, NHR.

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A stirred jacketed reactor was charged with: 1.0 g iodobenzene, 3 equivalents of triethylamine, 6 equivalents of ethanol, a catalytic amount of Pd(OAc)$_2$ and 2.0 g of a phosphonium ionic liquid. The reaction mixture was heated at
5 120° C for 14 hours under 5 bar CO partial pressure. After 14 hours, the reaction mixture was brought to room temperature, and the total reaction mixture was poured into water. The reaction mixture was extracted using petroleum ether (3x30 ml), the total organic phase was washed with water and concentrated
10 in vacuo followed by the distillation under reduced pressure. The ethylbenzoate product was recovered in nearly quantitative yield.

For these experiments, the phosphonium ionic liquid solvent selected from the group consisting of:

15   trihexyl(tetradecyl)phosphonium chloride;

       trihexyl(tetradecyl)phosphonium triflate;

       trihexyl(tetradecyl)phosphonium triflamide;

       trihexyl(tetradecyl)phosphonium bis-(2,4,4'-trimethylpentyl)phosphinate;

20   trihexyl(tetradecyl)phosphonium decanoate; and

       butylmethylimidazolium [bmin] hexafluorophosphate.

As shown in Table 9, all of the ionic liquids gave nearly quantitative yields of the required product material with the complete consumption of the starting iodobenzene. In
25 the case of trihexyl(tetradecyl)phosphonium decanoate, a mixture of products was obtained wherein 50 % of the product was a decanoate ester and 50 % was the expected ethyl ester; apparently the decanoate anion acts as a nucleophile and attacks the intermediate Ar-Pd-CO-X species.

33

These experiments demonstrate the particular suitability of the phosphonium phosphinate compounds of the current invention for palladium catalyzed carbonylation of aryl halides.

5

Table 9

Carbonylation of iodobenzene in various ionic liquids using Pd(OAc)2 catalyst

Exp.No.	Ionic Liquid	% Yield
1	trihexyl(tetradecyl)phosphonium chloride	100
2	trihexyl(tetradecyl)phosphonium triflate	97
3	trihexyl(tetradecyl)phosphonium triflamide	100
4	trihexyl(tetradecyl)phosphonium bis-(2,4,4-trimethylpentyl)phosphinate	100
5	trihexyl(tetradecyl)phosphonium decanoate	50+50
6	[bmin] hexafluorophosphate	78

Example 9: Bipyridine synthesis via the homo-coupling of bromopyridine in various ionic liquids using Pd(OAc)$_2$ catalyst

A stirred jacketed reactor was charged with:

1.0 g bromopyridine

2.0 g isopropanol

1.5 g K$_2$CO$_3$

0.03 g of Pd(OAc)$_2$

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The reaction mixture was heated at 120° C for 36 hours in a phosphonium ionic liquid solvent selected from the group consisting of:

trihexyl(tetradecyl)phosphonium triflate;

5   trihexyl(tetradecyl)phosphonium triflamide;

trihexyl(tetradecyl)phosphonium bis-(2,4,4'-trimethylpentyl)phosphinate;

trihexyl(tetradecyl)phosphonium dicyclohexylphosphinate; and

10   trihexyl(tetradecyl)phosphonium decanoate.

Overall yield of bipyridine using the different solvents was evaluated and is tabulated in Table 10. The yield of bipyridine varied considerably with choice of solvent. Good yields were obtained using phosphonium phosphinate compounds of the current invention as solvents, thus demonstrating the suitability of these compounds for use in bipyridine synthesis via the homo-coupling of bromopyridine in various ionic liquids using Pd(OAc)$_2$ catalyst.
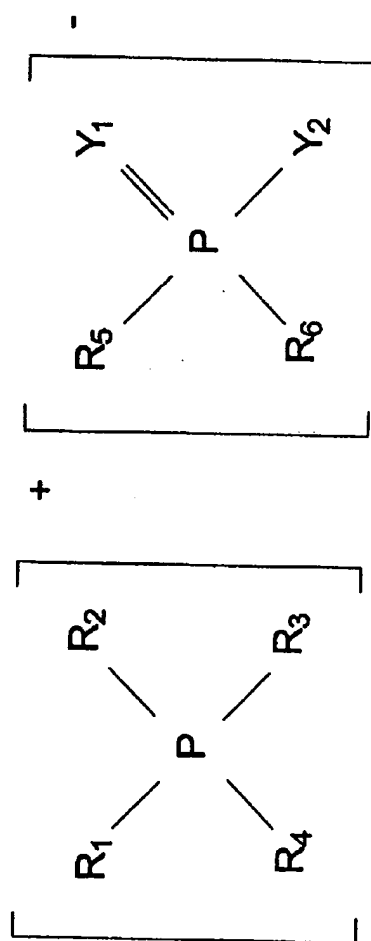
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Table 10

**Bipyridine synthesis via the homo-coupling of bromopyridine
using Pd(OAc)2 catalysis in various phosphonium ionic liquids**

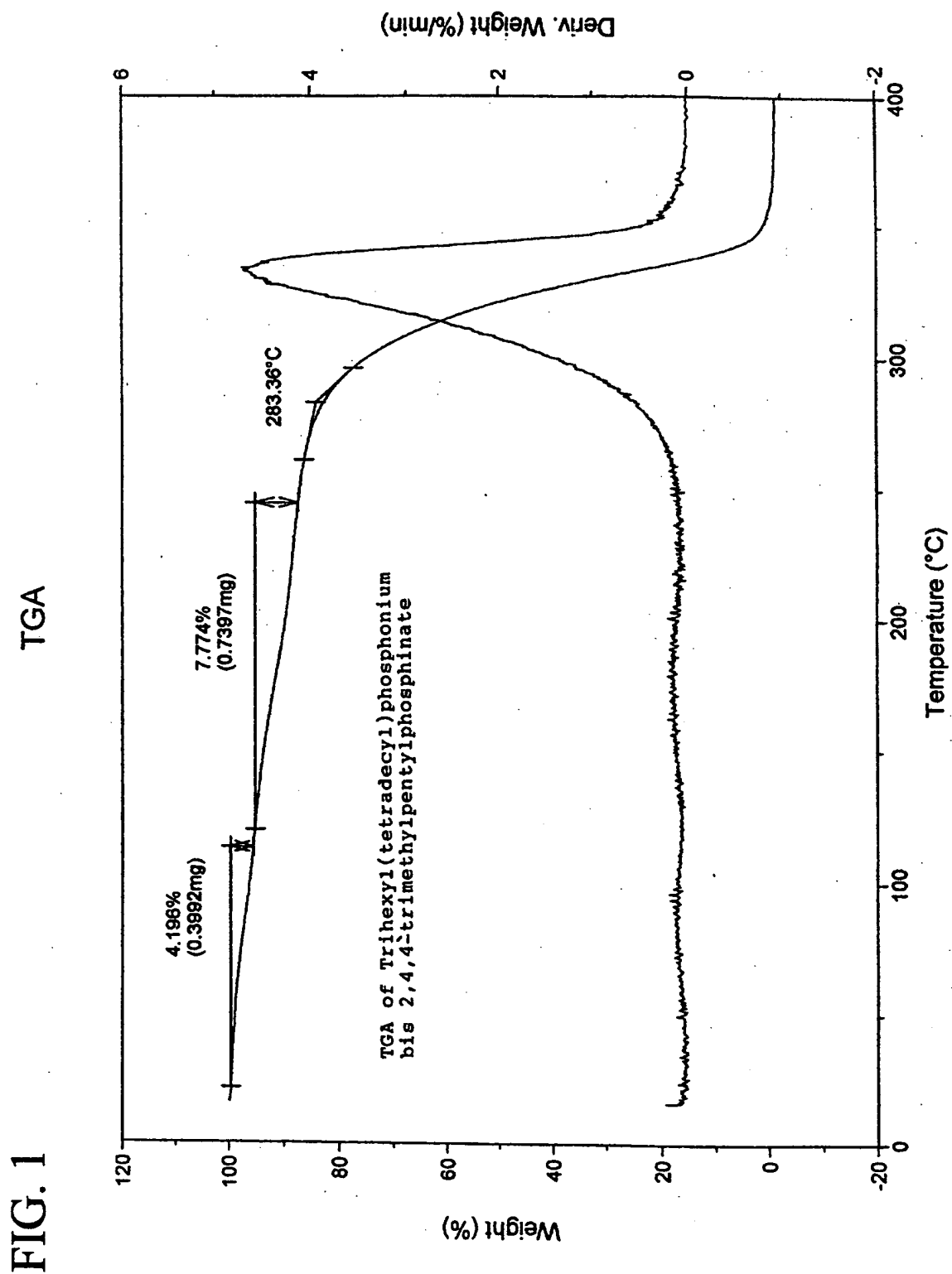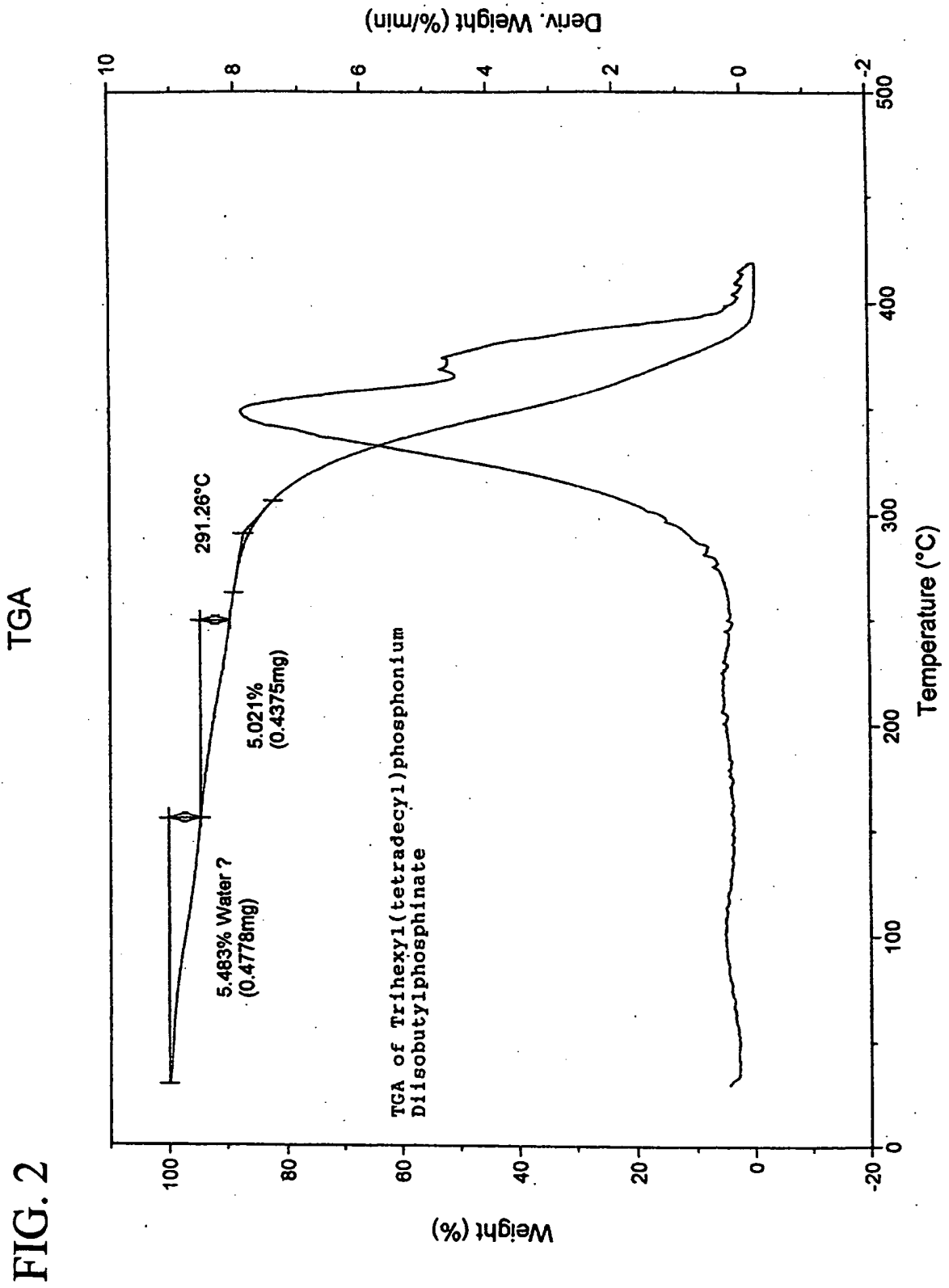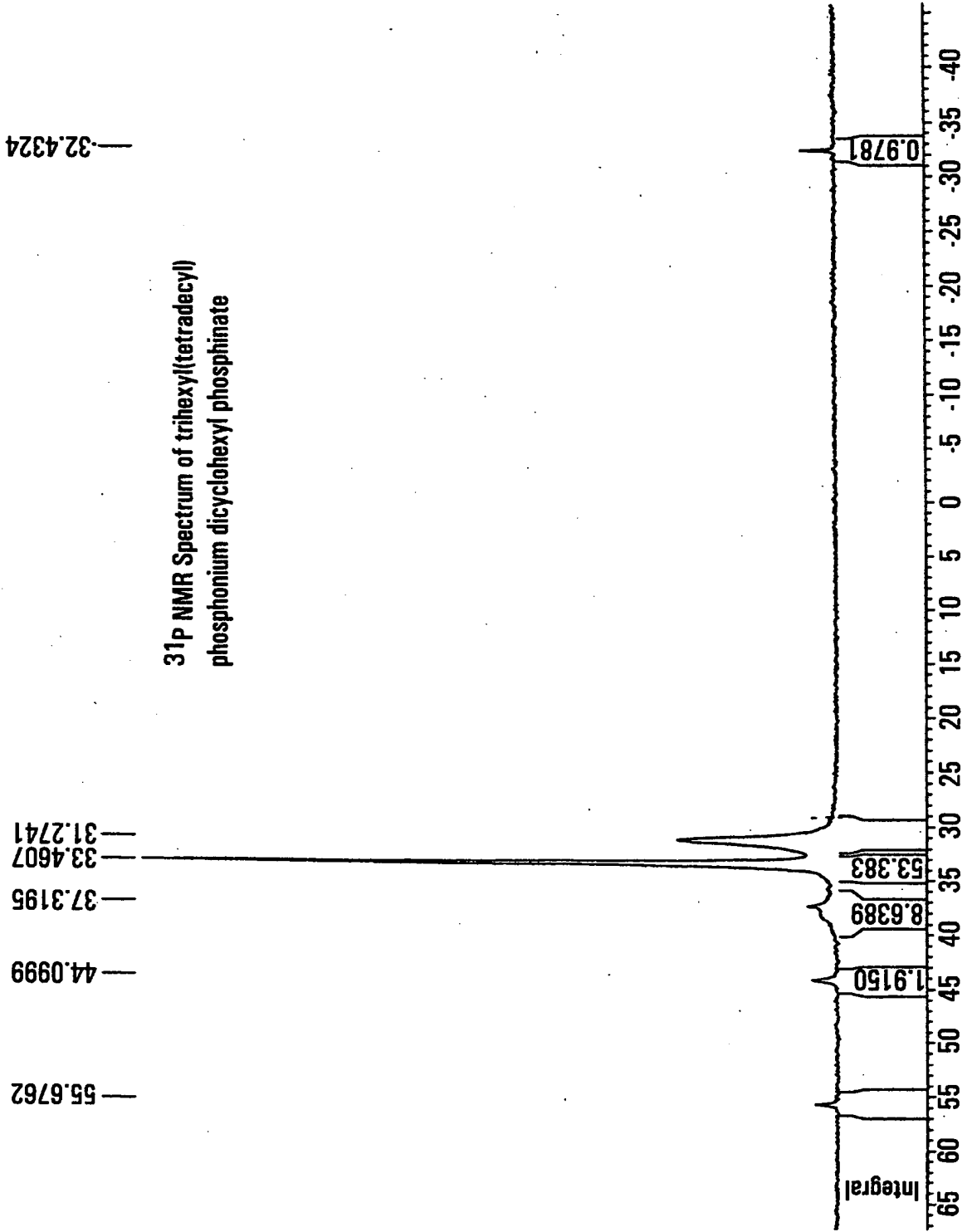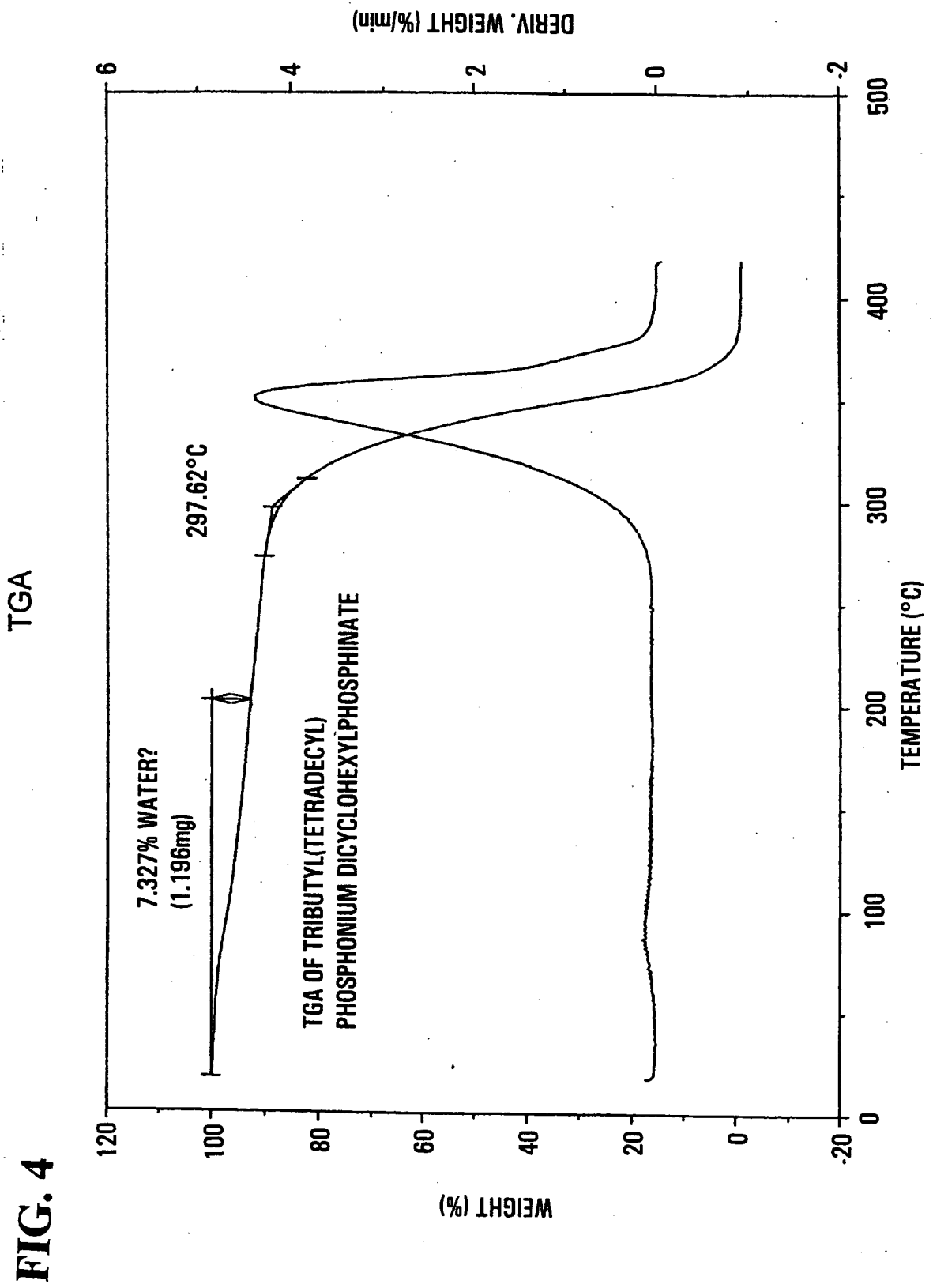|	Exp.No.	Ionic Liquid	% Yield
5	1	trihexyl(tetradecyl)phosphonium dicyclohexylphosphinate	52
	2	trihexyl(tetradecyl)phosphonium decanoate	100
	3	trihexyl(tetradecyl)phosphonium bis-(2,4,4-trimethylpentyl)phosphinate	52
10	4	trihexyl(tetradecyl)phosphonium triflamide	43
	5	trihexyl(tetradecyl)phosphonium triflate	36

$$\left[\begin{array}{c} R_2 \quad\ R_3 \\ \diagdown\ \diagup \\ P \\ \diagup\ \diagdown \\ R_1 \quad\ R_4 \end{array} \right]^{+} \left[\begin{array}{c} Y_1 \quad\ Y_2 \\ \diagdown\ \diagup \\ P \\ \diagup\ \diagdown \\ R_5 \quad\ R_6 \end{array} \right]^{-}$$

(I)

FIG. 1

TGA

TGA of Trihexyl(tetradecyl)phosphonium
bis 2,4,4-trimethylpentylphosphinate

FIG. 2

TGA

TGA of Trihexyl(tetradecyl)phosphonium
Diisobutylphosphinate

5.483% Water ?
(0.4778mg)

5.021%
(0.4375mg)

291.26°C

Weight (%)

Temperature (°C)

Deriv. Weight (%/min)

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FIG. 3

31P NMR Spectrum of trihexyl(tetradecyl)
phosphonium dicyclohexyl phosphinate

FIG. 4

TGA

DERIV. WEIGHT (%/min)

TEMPERATURE (°C)

WEIGHT (%)

297.62°C

7.327% WATER?
(1.196mg)

TGA OF TRIBUTYL(TETRADECYL)
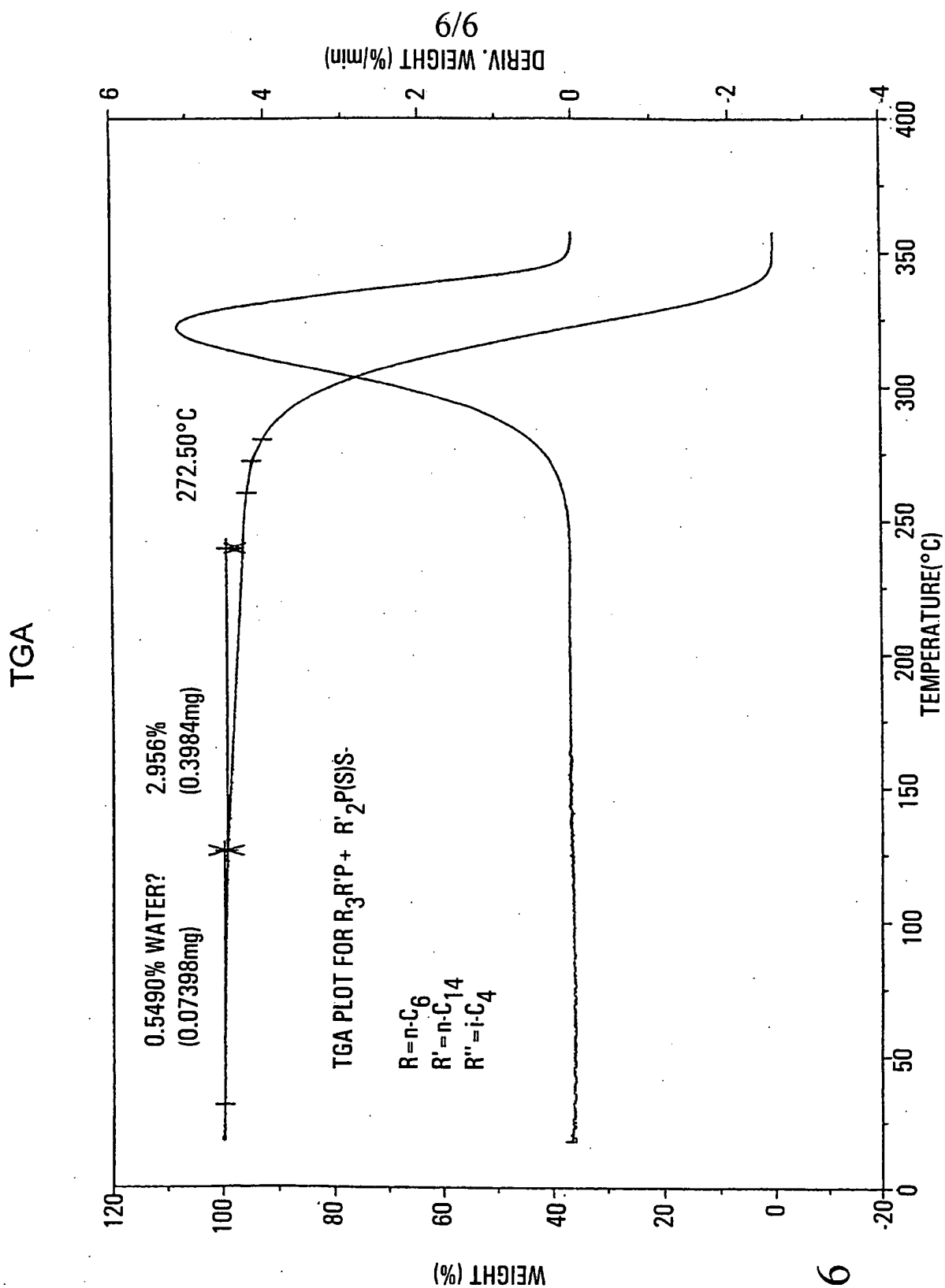PHOSPHONIUM DICYCLOHEXYLPHOSPHINATE

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FIG. 5

FIG. 6

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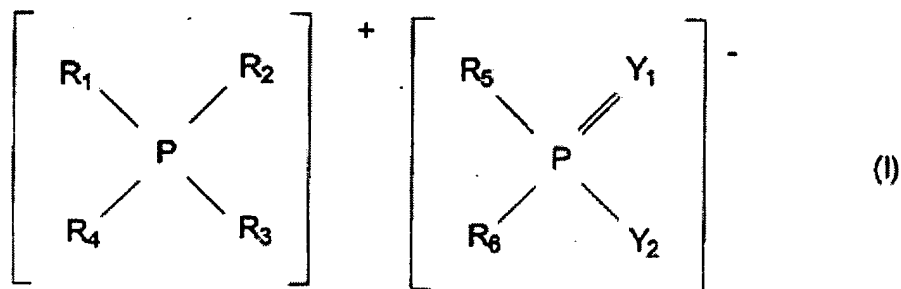
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(54) Titre : NOUVEAUX COMPOSES DE PHOSPHONIUM PHOSPHINATE ET ET LEURS PROCEDES DE PREPARATION
(54) Title: NOVEL PHOSPHONIUM PHOSPHINATE COMPOUNDS AND THEIR METHODS OF PREPARATION

(I)

(57) Abrégé/Abstract:
Novel phosphonium phosphinate compounds and their methods of preparation are disclosed. Phosphonium salts are ionic liquids that are useful as polar solvents. The novel phosphonium phosphinate compounds have the general formula (I): (see formula I) wherein R_1, R_2, R_3, R_4, R_5 and R_6 is independently a hydrogen or hydrocarbyl, unsubstituted or substituted; Y_1 is O or S; and Y_2 is O or S.

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